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## Progress at the Groningen AMS facility

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### Abstract

A new generation accelerator mass spectrometer (AMS) is operational since May 1994 at the Centre for Isotope Research in Groningen, The Netherlands. The fully automated and high throughput AMS system, manufactured by High Voltage Engineering Europa (HVEE) is dedicated to radiocarbon analysis. The model 4130  $^{14}\text{C}$  AMS is able to analyse up to 3000 samples per year. The system is characterized by a simultaneous transport of all three isotopes. Since the system is fully operational, various tests have been done to obtain insight in the system performance. Background, including sample preparation is, at present, approximately 45 ka and is still improving, whereas machine background is negligible. Performance tests on identical samples and cross checking with conventional  $^{14}\text{C}$  decay counting and  $^{13}\text{C}$  spectrometry showed a precision on the  $^{13}\delta$  and the  $^{14}\text{C}/^{12}\text{C}$  ratio of better than 2‰ and 0.5 pMC respectively.

### 1. Introduction

Accelerator mass spectrometry has established itself as an important dating tool in archaeology, geosciences, oceanography and other related research fields as well as biomedical applications. The ability to measure samples 1000 times smaller than used in scintillation or gas counting techniques in conjunction with high throughput widens the applications of AMS in the future [1].

At the Centre for Isotope Research in Groningen low background measurements by decay counting ( $^{14}\text{C}$  and  $^3\text{H}$ ) and stable isotope mass spectrometry ( $^2\text{H}$ ,  $^{13}\text{C}$ ,  $^{15}\text{N}$ ,  $^{18}\text{O}$ ) are used for many years in the research fields mentioned above. Results of the first project (Wiggle-match dating of Holocene raised peat bog deposits) will be published shortly [2].

In this facility one of the new generation high precision and high throughput  $^{14}\text{C}$  AMS systems has been put into routine operation since May 1994.

The system is built by High Voltage Engineering Europa and it is, after the Woods Hole machine [3], the second spectrometer which measures all three carbon isotopes simultaneously. This guarantees that all three isotopes are measured under the same conditions and furthermore it provides a strong, on-line diagnostic tool for the health of the complete system. The system is capable to date up to 3000 samples per year.

In earlier publications we reported on the individual performance of various machine sections [4,5]. In this article we mainly concentrate on overall system performance. Furthermore we will present some further developments on the 4130  $^{14}\text{C}$  AMS spectrometer.

### 2. System description

A detailed system description has been given in several publications [3–6]. Fig. 1 shows the layout of the system.

The cesium sputter type ion source is discussed elsewhere [7], whereas in Section 3 we describe the sample movement in detail. The source produces a  $\text{C}^-$  beam of typically 50  $\mu\text{A}$  at approximately 36 keV that is focussed by an einzel lens on a defining aperture. After this aperture the beam enters a recombinator [8] that consists of four second order corrected  $45^\circ$  magnets. The first pair of magnets analyses the beam and separates the desired masses 12, 13 and 14 at the symmetry plane of the recombinator by about 20 mm. Moveable beam stoppers in the symmetry plane allow every isotope to be injected and analysed separately. A chopper wheel located in the symmetry plane of the recombinator reduces the  $^{12}\text{C}$  beam load for the accelerator and the high energy spectrometer by a factor of about 90 [6]. The second pair of magnets recombine the three isotopes on the optical axis of the accelerator for simultaneous injection.

In the Tandetrion<sup>™</sup> accelerator the singly charged negative ions are focused and accelerated to a terminal, which operates at +2.5 MV. After charge changing to the mean

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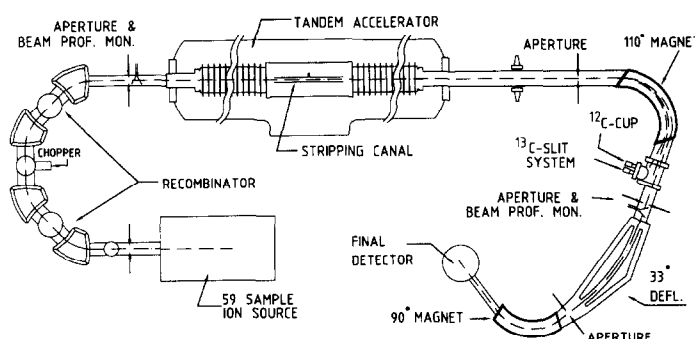


Fig. 1. Schematic layout of the HVEE  $^{14}\text{C}$  AMS system.

charge state  $3+$ , the ions are accelerated back to ground potential to an energy of 10 MeV. The high energy acceleration tube has a specially designed, inclined-field electrode arrangement to suppress ambiguities of ions with the same mass energy product as the wanted 10 MeV  $^{14}\text{C}^{3+}$  particles, which result from charge exchange processes during the acceleration to ground potential [6].

The final analysis is done by an achromatic high energy spectrometer, consisting of a  $110^\circ$  magnet, a  $33^\circ$  electrostatic deflector and a  $90^\circ$  magnet. The first magnet selects the  $3+$  charge state and separates the three isotopes. The  $^{12}\text{C}$  and  $^{13}\text{C}$  beams are measured in individual Faraday cups, located in the image point of the  $110^\circ$  magnet. Typical currents are in the range of 300 to 800 nA. The  $^{13}\text{C}$  cup is equipped with an integrated slit system [4]. The error signal that is generated by this system is fed back to the terminal voltage power supply and guarantees a stable beam position in the high energy spectrometer at any time. Because the slit system provides information on the beam position with a resolution of approximately 0.1 mm, it is also a strong diagnostic tool for tuning the system.

After the  $110^\circ$  magnet the  $^{14}\text{C}$  particles subsequently pass two additional dispersive elements, a  $33^\circ$  electrostatic deflector, which blocks almost all residual ambiguities with the same mass energy product as 10 MeV  $^{14}\text{C}^{3+}$ , and a  $90^\circ$  magnet for further background reduction, introduced by scattering effects at the  $33^\circ$  deflector plates or by two step charge exchange processes with the residual gas. The latter background is minimized by vacuum conditions below  $10^{-8}$  mbar in the  $^{14}\text{C}$  spectrometer.  $^{14}\text{C}$  ions are detected in an isobutane filled ionization chamber isolated from the vacuum system by a mylar foil ( $\sim 350 \mu\text{g}/\text{cm}^2$  thickness). This detector provides a  $dE/dx$  and a  $E_{\text{final}}$  signal. By gating these signals with standard electronic devices all unwanted particles are eliminated from the spectrum.

### 3. The ion source

As cratering of the target sample surface will cause increasing mass fractionation with time it should be mini-

mized if one wants to perform high precision measurements. This can be done by systematically moving the sample under the bombarding cesium beam such that the surface is sputtered more uniformly.

In the ion source of the model 4130  $^{14}\text{C}$  AMS system a computer controlled  $X$ - $Y$  sample movement is incorporated and is implemented in the software of the data acquisition. Up to 64 positions on the sample surface can be selected and the data acquisition is performed in time blocks of approximately 30 s. After every block, data are saved and the  $X$ - $Y$  table moves the sample to the next point on the sample surface to be irradiated by the cesium beam, thereby minimizing cratering effects. An additional advantage of this scanning of the sample is that the target material can be sputtered more effectively, that is up to the point where almost no material is left on the sample holder.

The diameter of the cesium beam on the sample surface can be controlled by the distance of the target holder to the ionizer, which can be set from the computer. Experiments have shown that this diameter can be adjusted down to approximately  $350 \mu\text{m}$ , allowing precise control of the sputtering cesium beam on the sample. Fig. 2 shows a picture of a sample after irradiation by the cesium beam on 5 different spots.

The ion source can accommodate up to 59 samples. In order to minimize cross contamination from sample to sample, the targets are stored in a revolving wheel, approximately 100 mm away from the sample under analysis.

## 4. System performance

System performance will be shown in 3 sections: 1) background measurements, 2) acceptance test performance obtained from ANU-sucrose targets and 3) comparison with measurements from  $^{13}\text{C}$  mass spectrometry and  $^{14}\text{C}$  decay counting (conventional).

### 4.1. Background measurements

The background level is, at present, 45 ka or around 0.2 pMC [9]. This includes target preparation, which is clearly

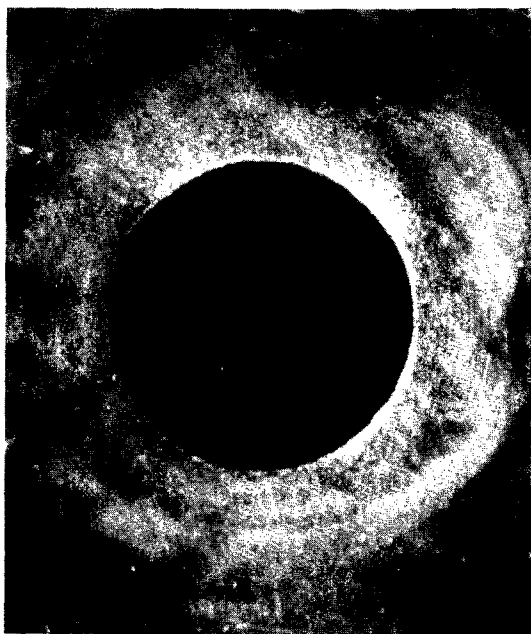


Fig. 2. Picture of a sample after irradiation on five different spots. The black area represents the 2 mm diameter graphite sample material.

the limiting factor. Since the first weeks of operation this number is still improving. Preparations to make the analysis of old samples possible (such as a semi-clean room etc.) are under way.

In order to determine machine background, a carbon free aluminum target ("machine blank") was analyzed. Fig. 3 shows the spectrum obtained. The peak at channel 100 is generated by alpha particles from a  $^{241}\text{Am}$  source, which is placed in the detector for testing purposes. The counts beyond channel 120 can be considered as background from the AMS system. In this spectrum only one count is observed in the  $^{14}\text{C}$  window, which indicates that machine background is negligible. In the top of Fig. 3 a (gated) spectrum from a ANU-sucrose target is shown. The window that is indicated was set by the detector electronics.

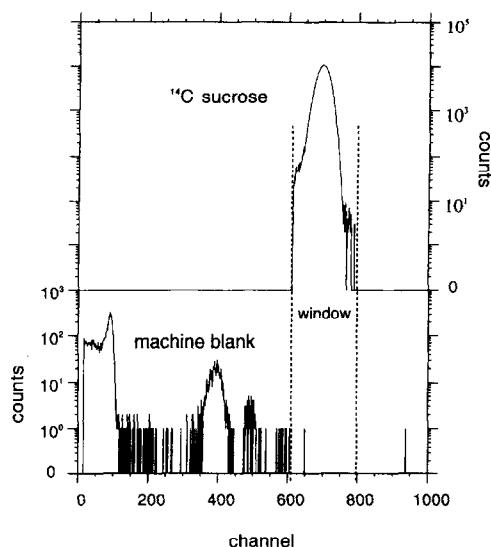


Fig. 3. Top: energy spectrum (gated), measured from a ANU-sucrose target. Bottom: energy spectrum (ungated), measured from a machine blank. Both spectra were counted for about 30 min.

#### 4.2. Acceptance test performance

In May 1994 the system was accepted based on the following tests. On two succeeding days a batch of identically produced ANU-sucrose samples was analyzed. Fig. 4 shows the  $^{13}\text{C}/^{12}\text{C}$  ratio as was measured over an interval of more than 2 hours on the first day. Six sucrose samples are used in 12 runs, whereby every sample was loaded twice. Every data point in the graph represents the mean of 80 current measurements carried out in 40 s. It can be seen that there is no long term drift on a ‰ level. Note that all data points are within 4‰. The small drifts that are visible for each individual run are most likely related to sample heat up introduced by the cesium sputter beam. To avoid these drifts an improved target cooling clamp has been installed and is presently under investigation.

Table 1 shows the mean and the standard deviation of measurements, performed on the two succeeding days. The

Table 1

Results from measurements of the two succeeding days. The  $^{12}\text{C}$  and  $^{13}\text{C}$  currents were in the range of 500 to 650 nA on both days

Sucrose 150.61 pMC	$^{13}\text{C}/^{12}\text{C}$		$^{14}\text{C}/^{13}\text{C}$		$^{14}\text{C}/^{12}\text{C}$		
	mean ( $\times 10^{-2}$ )	$\sigma_{\text{rel}}$	mean ( $\times 10^{-10}$ )	$\sigma_{\text{rel}}$	mean ( $\times 10^{-12}$ )	$\sigma_{\text{rel}}$	$\sigma$ (pMC)
First day, 6 samples, 2.2‰ statistics	1.0719	0.5‰	1.4705	1.7‰	1.5762	1.5‰	0.23
Second day, 5 samples, 1.7‰ statistics	1.0696	1.8‰	1.4718	1.8‰	1.5743	3.1‰	0.48
Difference between the means of both days	2.1‰		0.9‰		1.2‰ (0.18 pMC)		

counting statistics on the two days were 2.2‰ and 1.7‰ respectively. The ion source and the terminal voltage were switched off between both days. No data block was rejected and only a simple software window for the  $^{14}\text{C}$  counts from the  $E_{\text{final}}$  detector was used. The table shows that a precision below 0.5 pMC can be obtained on a routine basis. The last row in Table 1 present the difference between the means of both days. During the performance tests a day to day stability below 0.5 pMC was achieved.

#### 4.3. Comparison with $^{13}\text{C}$ spectrometry and $^{14}\text{C}$ decay counting

In the first weeks after the acceptance of the machine, extensive tests were performed on targets with a well known carbon isotopic ratio. The 12 samples that were used partly originated from IAEA intercomparison standards and partly they were submitted to the “conventional” laboratory at the University of Groningen. The  $^{14}\text{C}$  content in the samples varied from approximately 4 to 151 pMC. Fig. 5 shows the difference in the  $^{13}\delta$  and the  $^{14}\text{C}/^{12}\text{C}$  ratio between the results obtained from  $^{13}\text{C}$  spectrometry and  $^{14}\text{C}$  decay counting on one side (“conventional”) and AMS on the other as a function of the  $^{14}\text{C}$  content. In the figure dashed lines show the standard deviation in the measurements, which is 1.5‰ for  $^{13}\delta$  and 0.4 pMC for the  $^{14}\text{C}/^{12}\text{C}$  ratio. These results are comparable to those obtained during the acceptance test (Table 1) and show that the overall system performance on  $^{13}\delta$  and the  $^{14}\text{C}/^{12}\text{C}$  ratio is better than 2‰ and 0.5 pMC respectively.

#### 5. Further developments

The next HVEE  $^{14}\text{C}$  spectrometer, purchased by the University of Kiel, Germany has been assembled and is

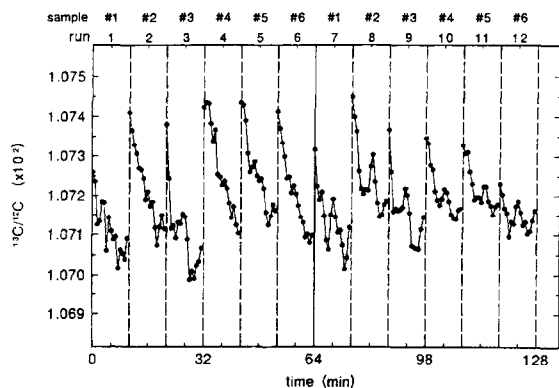


Fig. 4. Stability measurement of the  $^{13}\text{C}/^{12}\text{C}$  ratio over 2 hours for 6 different ANU-sucrose samples.

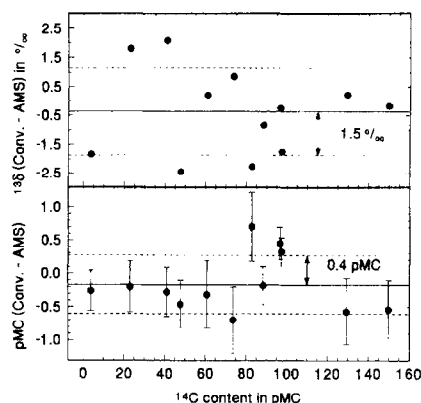


Fig. 5. Comparison of  $^{13}\delta$  and the  $^{14}\text{C}/^{12}\text{C}$  ratio between “conventional” and AMS measurements for 12 samples with varying  $^{14}\text{C}$  content. Dashed lines indicate the standard deviation.

currently in its final test phase. In consultation with the University of Kiel several features have been added:

- Both acceleration tubes have been modified and are now fully magnetically suppressed. First tests at rated voltage and normal beam load indicated that the radiation level is now at such a level as to allow full access to the machine during operation.
- Two beam profile monitors were added to the system (see Fig. 1). The monitors are integrated in the computer control system and the beam profiles can be displayed on the computer screen.
- An aperture between the  $33^\circ$  electrostatic deflector and the  $90^\circ$  magnet has been added.

The last two features are very useful diagnostic tools for the operator during tune-up of the system. The beam profile monitors allow the system to be tuned in such a way that the position of the beam waists are in correspondence to the optical design. By the addition of the earlier mentioned aperture the tuning of the  $33^\circ$  electrostatic deflector and the  $90^\circ$  magnet is uncoupled and therefore straightforward.

#### 6. Conclusions

The fully automated and high throughput HVEE  $^{14}\text{C}$  AMS system at the University of Groningen is fully operational. In the ion source, motor controlled sample movements give full control over the sputtering process and therefore mass fractionation resulting from sample cratering is minimized. The system features a simultaneous transport and analysis of all three carbon isotopes which is a strong on-line diagnostic tool for the health of the entire system. Various measurements have shown that machine background is negligible and that the overall system performance on  $^{13}\delta$  and the  $^{14}\text{C}/^{12}\text{C}$  ratio is better than 2‰ and 0.5 pMC respectively.

Further developments on the next HVEE  $^{14}\text{C}$  spectrometer, shortly to be shipped to the University of Kiel, simplify the tuning of the machine.

### Acknowledgements

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